AD

INVESTIGATING AND SUPPLYING HALID FLUX-GROWN KTP CRYSTALS

Final Technical Report

by

Dominique LUPINSKI

July 1995

United States Army
EUROPEAN RESEARCH OFFICE OF THE U.S. ARMY

London England

CONTRACT NUMBER DAJA45-93-C-0048

CRISTAL LASER S.A.

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Therefore, the experiments were performed on crystals from Cristal Laser S.A. The main tests provided for in the contract were performed by qualified scientific personal at Cristal Laser. However, some experiments which required specific equipment had to be realized in external laboratories.

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The 9 AR coated KTP and the 32 mirrors were supplied with the specified control data.

14. SUBJECT TERMS

KTP crystals for OPO : quality control, optical absorption, homogeneity.

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Our principal Investigator on this program was Dominique LUPINSKI, but we also include in our list of people to thank Professor Gérard MARNIER, of the Université de Dijon in France, who lent considerable expert advice, Philippe VILLEVAL, who actually grew the crystals, Christophe BONNIN, who processed them, and Hervé ALBRECHT, who analysed them and made sure they met requirements. We also wish to thank the German firm that produced the mirrors,and in particular, Dr. EBBERT,and also the French firm SFIM ODS Mr GEENEN, with whom we interacted during this program.

Finally it should be added that this program was a vital testimony to the success which we can achieve when mutual US-French technical goals are pursued.

ABSTRACT

The original purpose of this contract was to supply $KTiOPO_4$ (KTP) crystals grown by Cristal Laser S.A.by flux of alcali metal halide.

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KEY-WORDS

$KTiOPO_4$

KTP

Halide flux-grown

OPO

Absorption

Transparency

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1. INTRODUCTION

1.1. Presentation of the contractor

CRISTAL LASER S.A. is specialized in the growth and processing of inorganic nonlinear crystals and performs also studies and special cuts according to the users' requirements.

The company operates under exclusive licence international CNRS patents[1] regarding the synthesis of $KTiOPO_4$ and isotypes by flux of alcali metal halide. These patents concretize the research carried out since 1983 by the team of G. Marnier at the Université de Nancy, France, which were transferred to the Université de Bourgogne in 1994.

1.2. Growth of KTP at Cristal Laser

Potassium Titanyl Phosphate $KTiOPO_4$ is an orthorhombic crystal (mm2 space group), optically biaxial, transparent between 0.35 and 4.5 µm, non centrosymmetric and endowed with excellent properties for different nonlinear optical applications. It presents a strong second-order electrical susceptibility, a high optical damage threshold and wide angular and thermal acceptances.

The Second-Harmonic Generation Type II in KTP allows to generate radiations from 0.5 to 1.5 µm. Radiations in the field 1 → 4.5 µm can also be obtained by parametric interactions.

The crystals produced for this contract were grown in concentrated solutions of oxides and alcali metal halide raised to high temperature (600°C to 1000°C). This method allows to obtain a high crystalline perfection and low residual absorption (<0.1%/cm @ 1.064 µm).

1.3. Introduction to the quality controls at Cristal Laser

The quality criteria of nonlinear crystals are not easy to define. In fact, physical properties of the crystals are strongly correlated with their nonlinear optical properties. So the main problem is to determine the level of an acceptable quality according to the final application. For example, we could not yet define all the significant parameters for Optical Parametric Oscillation (OPO).

Our experience shows that, whatever the application, evaluating the quality level of KTP requires to define and control more than 10 basic criteria during the whole growth-process. Quality parameters such as homogeneity, dark-lines, impurities, optical absorption, transparency, ionic conductivity, thermal stability, extinction ratio, conversion efficicency, life-time, optical damage threshold, depend on the growth conditions :

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[1] CNRS-University of Nancy I licence - G. MARNIER, B. BOULANGER, B. MENAERT, M. METZGER. US patent n°4,746,396 (28/07/86) - US Patent n°4,961,819 (22/01/88).

stoechiometry, linear growth-speed, temperature of growth, global crystallization efficiency, rotation speed, thermal gradient, thermal shocks, etc...

Lastly a great difficulty is to make the measurements in absolute. Indeed the values which are obtained strongly vary according to the conditions of experimentation (wavelength, polarization of the laser beam, repetition rate, ...).

Regarding KTP as an Optical Parametric Oscillator, which is dealt with this report, we focused on two criteria which seem to be the most significant for this application : the optical absorption at 1.064 µm and the homogeneity.

2. LIST OF THE KTP DELIVERED WITHIN THE CONTRACT

Reference	Cutting angle		Size	AR coating	Reference of batch
	θ	ϕ			
34 00 203	51.5°	0°	5x6x10 mm³	1.064 + 2.128 µm	173/94/6+83/95/6
34 00 204	51.5°	0°	5x6x10 mm³	1.064 + 2.128 µm	173/94/6+83/95/6
34 00 206	51.5°	0°	5x6x1.73 mm³	1.064 + 2.128 µm	173/94/6+83/95/6
40 40 109	90°	0°	5x5x10 mm³	1.064 + 1.52-1.58 µm	65/95/6+79/95/6
44 00 205	90°	0°	5x5x10 mm³	1.064 + 1.52-1.58 µm	TM/270795
40 30 724	90°	0°	5x5x2 mm³	1.064 + 1.52-1.58 µm	65/95/6+79/95/6
40 40 221	90°	90°	5x5x10 mm³	1.064 + 1.52-1.58 µm	65/95/6+78/95/6
40 40 222	90°	90°	5x5x10 mm³	1.064 + 1.52-1.58 µm	65/95/6+78/95/6
34 00 209	90°	90°	5x5x2 mm³	1.064 + 1.52-1.58 µm	65/95/6+79/95/6

3. TESTS OF THE KTP

3.1. *Crystallographic orientation*

Performed by : C. BONNIN, Engineer, Cristal Laser.

The crystallographic orientation has been measured with a X-Ray goniometer ensuring ± 3' accuracy.
Before the polishing step the precision of the cutting angle reached ± 3' for X-cut and Y-cut and 12' for θ = 51.5°.

All the finished crystals have a cutting error ≤ 30'. The Z-face is also orientated with less than 30' accuracy.

3.2. *Flatness and parallelism*

Performed by : D. LUPINSKI, principal investigator, Cristal Laser
C. BONNIN, engineer, Cristal Laser.

Flatness is better than $\lambda/10$ @ 633 nm. PV values are typically 0.07 or 0.08 λ.
For example, the first surface of the crystal ref. 40 40 109 is $0.08 \pm 0.01\ \lambda$, including the edge effect. The PV value on the crystal center is below 0.05 λ for X-profile (appendix A, fig. 1, 2, 3).

The parallelism is below 10 arc seconds due to the double-side polishing technique used. Parallelism values reach 1 µm for 40 cm.

3.3. *Optical absorption*

Performed by : Dr. V. WILLAMOWSKI, Laser Zentrum Hannover, Germany
Dr. J. MANGIN, Université de Nancy, France

The principal effect of the optical absorption is a mismatch of the crystals which induces instabilities of the conversion efficiency.

The measurement in absolute is generally critical. It requires very accurate means of measurement because of the low absorption of the KTP, particularly at 1.064 µm (40-100 ppm).

Two ways of measurement have been investigated :

- The calorimetric method [2]
- The pyrospectrometric method [3]

At 1.064 µm, the values are quite similar with the both techniques, that is to say $\leq 0.05\%$/cm.

The absorption of a 5 mm long KTP cut in the SHG direction is respectively 70 and 80 ppm for the two polarizations Kxy and Kz.
With the calorimetric technique, all the crystals measured present an absorption below 0.02%/cm at 1.064 µm.
Seeing these results, the production of Cristal Laser is quite homogeneous. However absolute values of absorption have to be considered cautiously : indeed the very low values measured depend on the method and the control bench used. Hence a comparative

[2] M. Dieckmann, V. Willamowski, D. Ristau, H. Welling - Laser Zentrum Hannover e.V., *Antireflective Coatings on Optical Fibers for High Power Solid-State Lasers*,
[3] J. Mangin, A. Khodjaoui and G. Marnier, *Optical absorption of KTP single crystals*, Physica Status Solidi (a) 120 (1990) KIII.

study requires obviously the same control bench as well as strictly similar crystals (same size and same crystallographic orientation).

3.4. Transmission 0.3-5 µm

As for the absorption, the transmission was measured on KTP crystals of similar length, same crystallographic orientation, attending to precise the polarization of the laser beam. The crystals were measured before coating.

A great difference can be observed between 3.2 µm and 3.8 µm at the two polarizations. The idler is polarized along OZ for X-cut and Y-cut.

For X-cut : $\lambda i = 3.29$ µm : the transmission along OZ is higher than along OY.
For Y-cut : $\lambda i \approx 3.5$ µm : the transmission along OZ is lower than along OX.

The transmission along Y-cut seems generally lower than along X-cut

For equivalent direction and beam polarization, the transmission is quite the same with the different crystals. The transmission graph presents the same profile including the accuracy level of the measurement.

The absorption band at 2.8 µm (OH^- band) is low compared with other KTP sources, in particular hydrothermally grown crystals.

3.5. Homogeneity

In KTP crystals, inhomogeneities can be observed with different devices :

- Inclusions of very small diameter (HeNe laser can pass the crystals and scattering centers can be observed with a microscope)[4]

- Larger inclusions can be studied with birefringence interferometry. Along b axis, pictures show {011} and {100} inclusions[4].

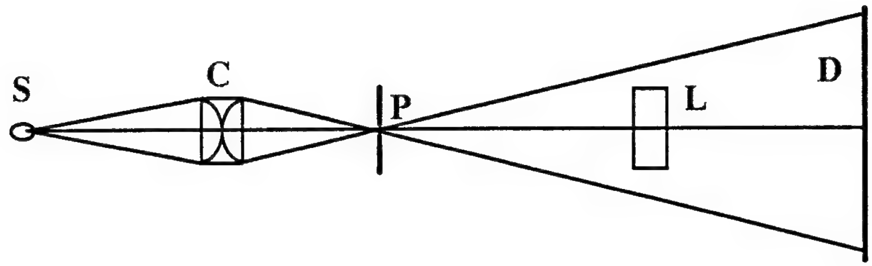
- Optical homogeneity has been evaluated by measuring wavefront distortion in transmission with circularly polarized wave. Variations of respectively 1.6 10^{-4} and 7.1 10^{-6} of nx and nxy refractive indices have been measured[5] .

- Variation of the nonlinear effects by scanning the crystals with a tight focused laser beam. The inhomogeneity of refractive indices may induce a variation of the phase-matching angle.

---

[4] R.J. Bolt, M.H. Van Der Mooren and H. De Hass, *Growth of KTP single crystals by means of Phosphate and Phosphate/Sulfate fluxes out of a three-zone furnace*, Journal of Crystal Growth 114 (1991), 141-152.
[5] T. Sasaki, A. Miyamoto, A. Yokotami and S. Nakai, *Growth and optical characterization of large potassium titanyl phosphate crystals*, Journal of Crystal Growth 128 (1993), 950-955.

- The shadow technique : The sample must be a fine polished cube with parallelism better than 10 arc seconds and flatness less than $\lambda/10$. A white light is used as a source with lenses to focus on a pinhole. The light is diffracted and goes through the crystal. The projection of the KTP crystal is stored with a fast, continuous tone, contrast graded, black and white paper placed behind the sample (see the figure).



S : Light source
C : Lenses
P : Pinhole
L : KTP crystal
D : Paper

When the sample is perfectly homogeneous, the outlines of the crystal can be observed with uniform color. On the contrary, when the crystal shows bulk local defects, the optical path is changed. On the paper, we note complex figures drawn as a variation of the grey tone. It is very difficult to correlate theoretical calculations with the experimental datas. This is the reason why we just compare all the different samples to estimate the quality.

4. AR COATINGS

For the OPO application, the KTP have to AR coated at the pump and at the signal wavelength. We selected SFIM ODS as a subcontractor, which performed the following AR coatings :

n°1 : dual-band AR coating : $\lambda p = 1.064$ µm
$\lambda s = 2.128$ µm
n°2 : dual-band AR coating : $\lambda p = 1.064$ µm
$1.52\ \mu m \leq \lambda s \leq 1.58\ \mu m$

Unfortunately the first face of the two batches were not at the maximum efficiency they could reach. The measurement was made directly on KTP for the two indices ny and nz.

AR COATING 1.064 µm + 2.128 µm			
Face	**Ref. batch**	**λ = 1.064 µm**	**λ = 2.128 µm**
Face n°1	173/94/6	1% ≤ R ≤ 1.23%	0.13% ≤ R ≤ 0.23%
Face n°2	83/95/6	Rny = 0.6% Rnz = 0.4%	Rny ≈ 0.1% Rnz = 0.1%

AR COATING 1.064 µm + 1.52-1.58 µm			
Ref. batch	**λ = 1.064 µm**	**λ = 1.52 µm**	**λ = 1.58 µm**
65/95/6	Rny = 1.00% Rnz = 1.35%	Rny = 0.35% Rnz = 0.60%	Rny = 1.20% Rnz = 1.65%
78/95/6	Rny = 0.15% Rnz = 0.30%	Rny = 0.50% Rnz = 0.80%	Rny = 0.50% Rnz = 0.70%
79/95/6	0.1% ≤ R ≤ 0.2%	0.4% ≤ R ≤ 0.55%	0.6% ≤ R ≤ 0.8%
TM/270795	R ≤ 0.1%	R ≤ 0.15%	R ≤ 0.15%

5. OPO MIRRORS

CaF2 mirrors for testing the KTP in the OPO configuration were provided by a german company which could meet the specifications and the budget of the contract.

Required specifications :

Substrate : CaF2 1" diameter, 1/4" thick			
	Surface #1	**Surface #2**	**Quantity**
M1	AR 1.064 µm	AR 1.064 µm HR 1.50-1.65 µm	4
M2	HR 1.064 µm 50% ± 10% @ 1.5-1.65 µm	AR 1.5-1.65 µm	4
M3	HR 1.064 µm 70% ± 10% @ 1.50-1.65 µm	AR 1.5-1.65 µm	4
M4	HR 1.064 µm 90% ± 10% @ 1.50-1.65 µm	AR 1.50-1.65 µm	4
M5	AR 1.064 µm	AR 1.064 µm HR 1.9-2.3 µm	4
M6	AR 1.064 µm 50% ± 10% @ 1.9-2.3 µm	AR 1.9-2.3 µm	4
M7	HR 1.064 µm 70% ± 10% @ 1.9-2.3 µm	AR 1.9-2.3 µm	4
M8	HR 1.064 µm	AR 1.9-2.3 µm	4

Reflection measurements :

Type of mirror	M1-M4 / M5-M8	1064 nm / 1064 nm	1500→1650 nm / 1900 → 2300 nm	1520 nm / 2100 nm	1570 nm / 2160 nm
M1	requested	T > 90%	T < 1%		
	measured	T = 92%	T < 0.5%	T = 0.3%	T = 0.3%
M2	requested	T < 1%	40% ≤ T ≤ 60%		
	measured	T < 1%	37% ≤ T ≤ 68%	T = 58%	T = 40%
M3	requested	T < 1%	20% ≤ T ≤ 40%		
	measured	T < 1%	23% ≤ T ≤ 55%	T = 48%	T = 28%
M4	requested	T < 1%	0% ≤ T ≤ 20%		
	measured	T < 1%	5% ≤ T ≤ 12%	T = 9%	T = 5%
M5	requested	T > 90%	T < 1%		
	measured	T = 89%	T < 1%	T < 1%	T < 1%
M6	requested	T < 1%	40% ≤ T ≤ 60%		
	measured	T < 1%	34% < T < 58%	T = 40%	T = 35%
M7	requested	T < 1%	20% ≤ T ≤ 40%		
	measured	T < 1%	18% < T < 32%	T = 21%	T = 18%
M8	requested	T < 1%	0% ≤ T ≤ 20%		
	measured	T < 1%	8% ≤ T ≤ 16%	T = 12%	T = 10%

APPENDIX # 1

Flatness data

4040109F1 00:09 10/31/94 T P

rms: 0.010
wv: 633.0nm

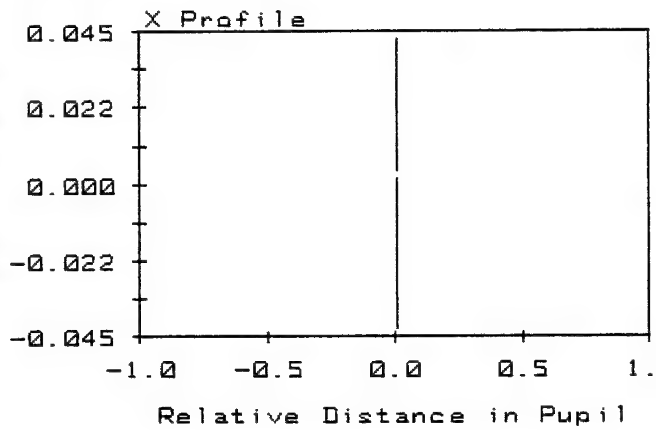
OPD

p-v: 0.084
pupil: 90%

0.039	0.017
0.033	0.011
0.028	0.005
0.022	-0.000
0.017	-0.006

x = 125, 0.00
y = 128, -0.00
Height = 0.003

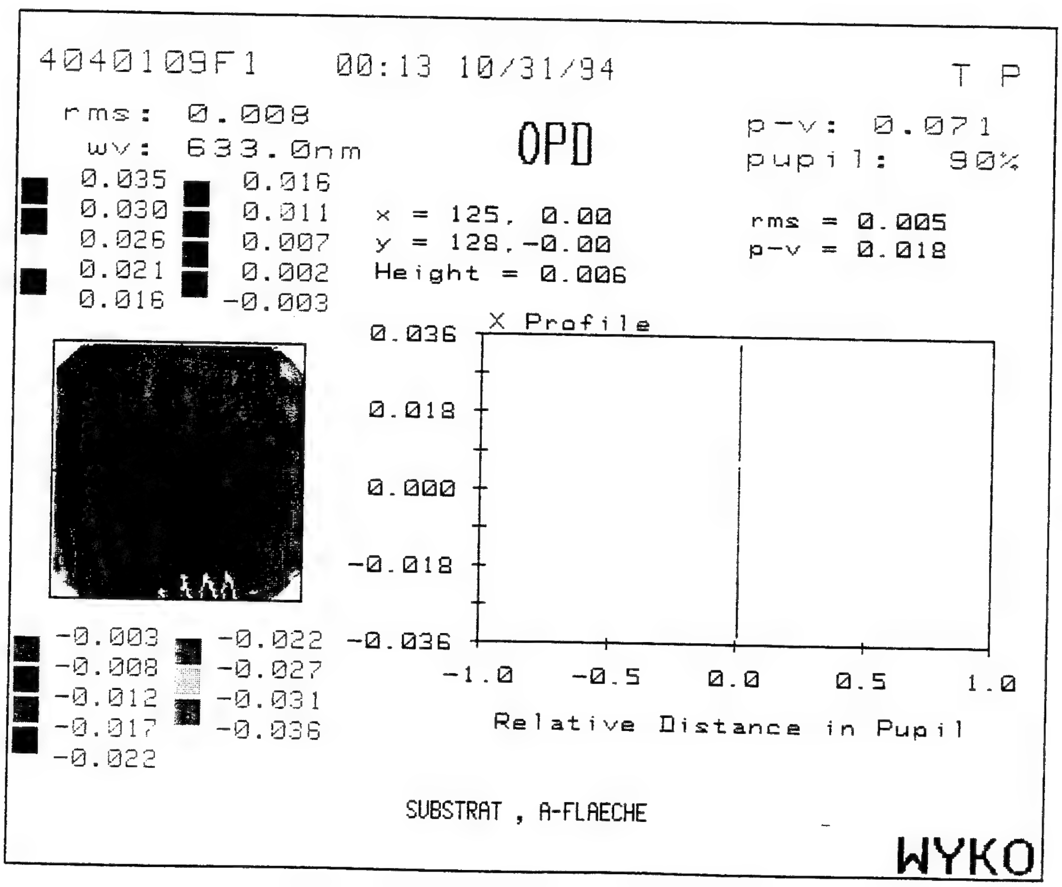
rms = 0.007
p-v = 0.033



-0.006	-0.028
-0.011	-0.034
-0.017	-0.039
-0.023	-0.045
-0.028	

SUBSTRAT , A-FLAECHE

WYKO



4040109F1 00:13 10/31/94
T P
rms: 0.008
wv: 633.0nm
OPD
p-v: 0.071
pupil: 90%
0.035
0.030
0.026
0.021
0.016
0.016
0.011
0.007
0.002
-0.003
x = 125, 0.00
y = 128, -0.00
Height = 0.006
rms = 0.005
p-v = 0.018
X Profile
0.036
0.018
0.000
-0.018
-0.036
-1.0
-0.5
0.0
0.5
1.0
Relative Distance in Pupil
-0.003
-0.008
-0.012
-0.017
-0.022
-0.022
-0.027
-0.031
-0.036
SUBSTRAT , A-FLAECHE
WYKO

4040109F2 00:33 10/31/94 T P

rms: 0.022
wv: 633.0nm

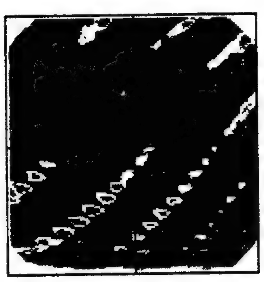
OPD

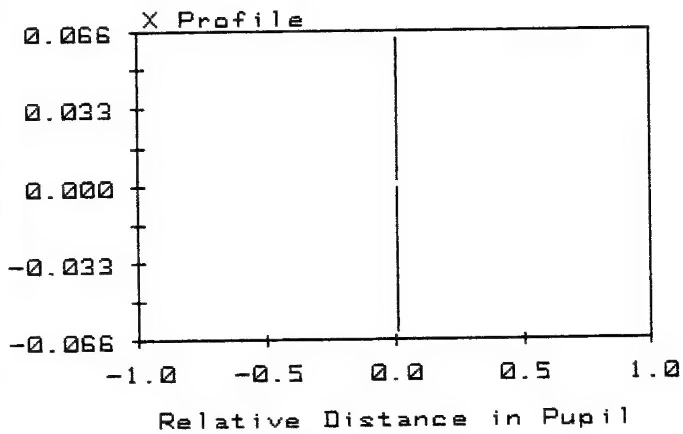
p-v: 0.120
pupil: 90%

0.054	0.022
0.046	0.014
0.038	0.006
0.030	-0.002
0.022	-0.010

x = 125, 0.00
y = 127, 0.00
Height = -0.002

rms = 0.020
p-v = 0.065





-0.010	-0.042
-0.018	-0.050
-0.026	-0.058
-0.034	-0.066
-0.042	

SUBSTRAT , A-FLAECHE

WYKO

APPENDIX # 2

Absorption data

KTP 40 30 71 07

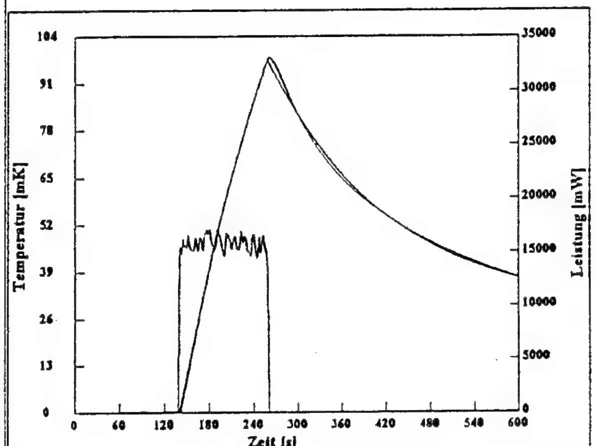
KTP C

uncoated
z - polarized

Protocol of Absorption
Date: 13.06.1994
Wavelength: 1064 nm

Name of File: 27054C

Absorption : 70.9 ppm



Laser power	15870	mW
Time of irradiation	120	s
Substrate material	KTP	
Mass of substrate	140	mg
Specif. heat	0.729	J/gK
Calibration factor	1.4	
Exponential method	70.89	ppm
Gradient method	67.47	ppm
Integral method	69.38	ppm
Extrapolation method	62.93	ppm
Loss coeff. during irradiatio	-21.56	1/h
Loss coeff. after irradiation	-21.56	1/h
Temp.- Drift	0	mK/h
Temp.- Offset	0	mK
Temp.- Plateau	0	mK

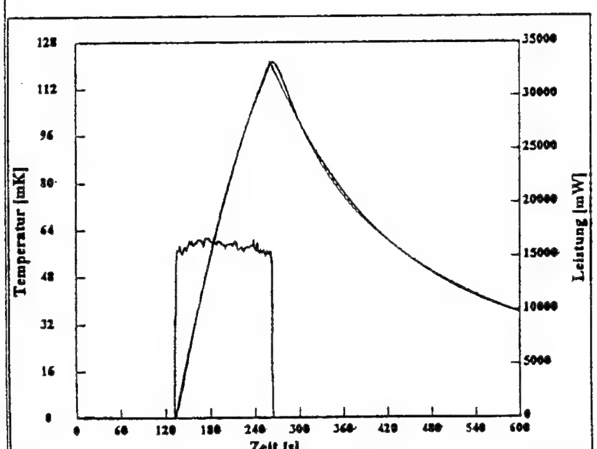
KTP C

uncoated
xy - polarized

Protocol of Absorption
Date: 13.06.1994
Wavelength: 1064 nm

Name of File: 27054D

Absorption : 82.7 ppm



Laser power	16090	mW
Time of irradiation	130	s
Substrate material	KTP	
Mass of substrate	140	mg
Specif. heat	0.729	J/gK
Calibration factor	1.4	
Exponential method	82.66	ppm
Gradient method	79.38	ppm
Integral method	81.71	ppm
Extrapolation method	75.04	ppm
Loss coeff. during irradiation	-21.6	1/h
Loss coeff. after irradiation	-21.6	1/h
Temp.- Drift	0	mK/h
Temp.- Offset	0	mK
Temp.- Plateau	0	mK

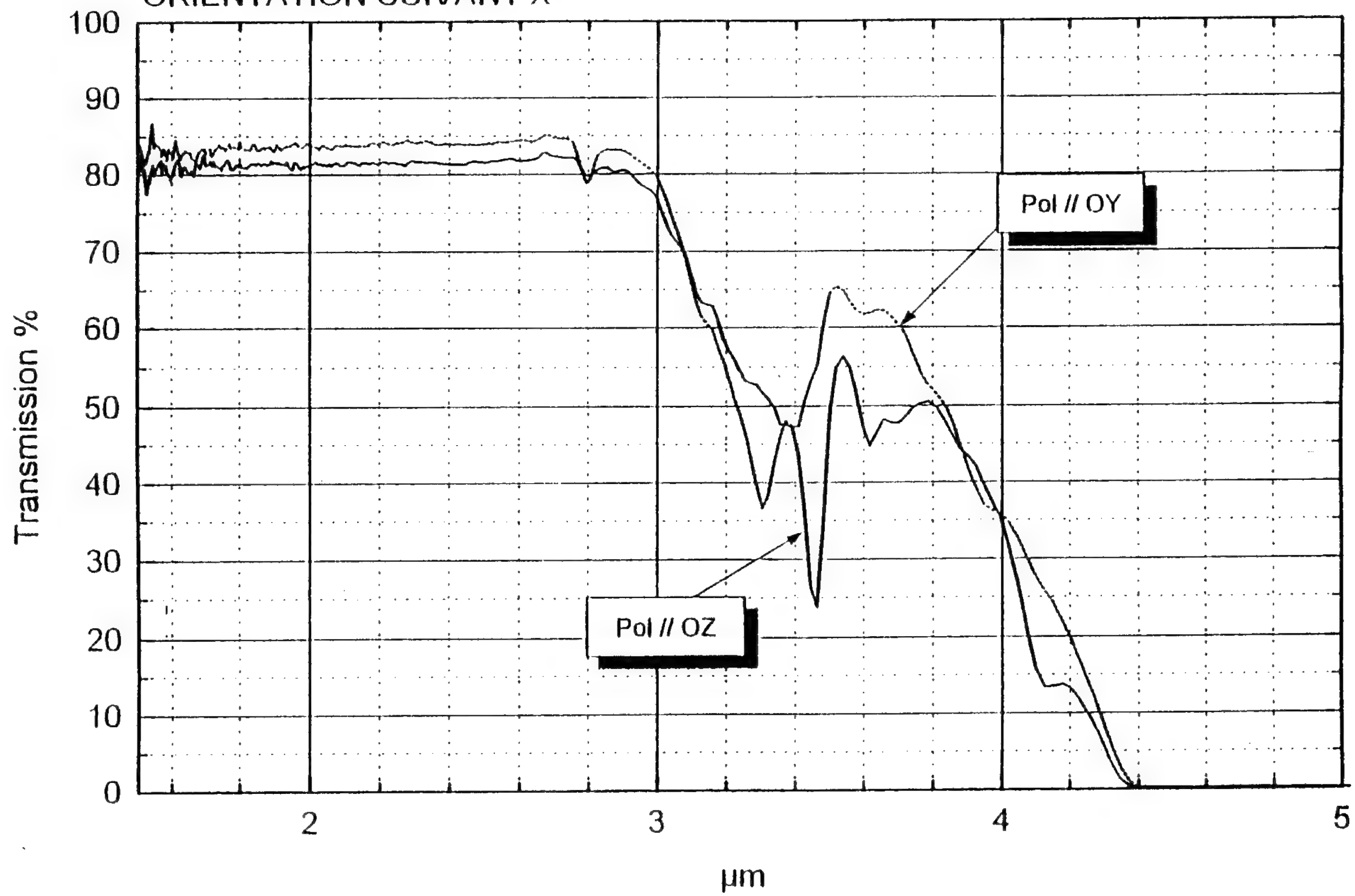
5 MM

APPENDIX # 3

Transmission graphes of KTP crystals

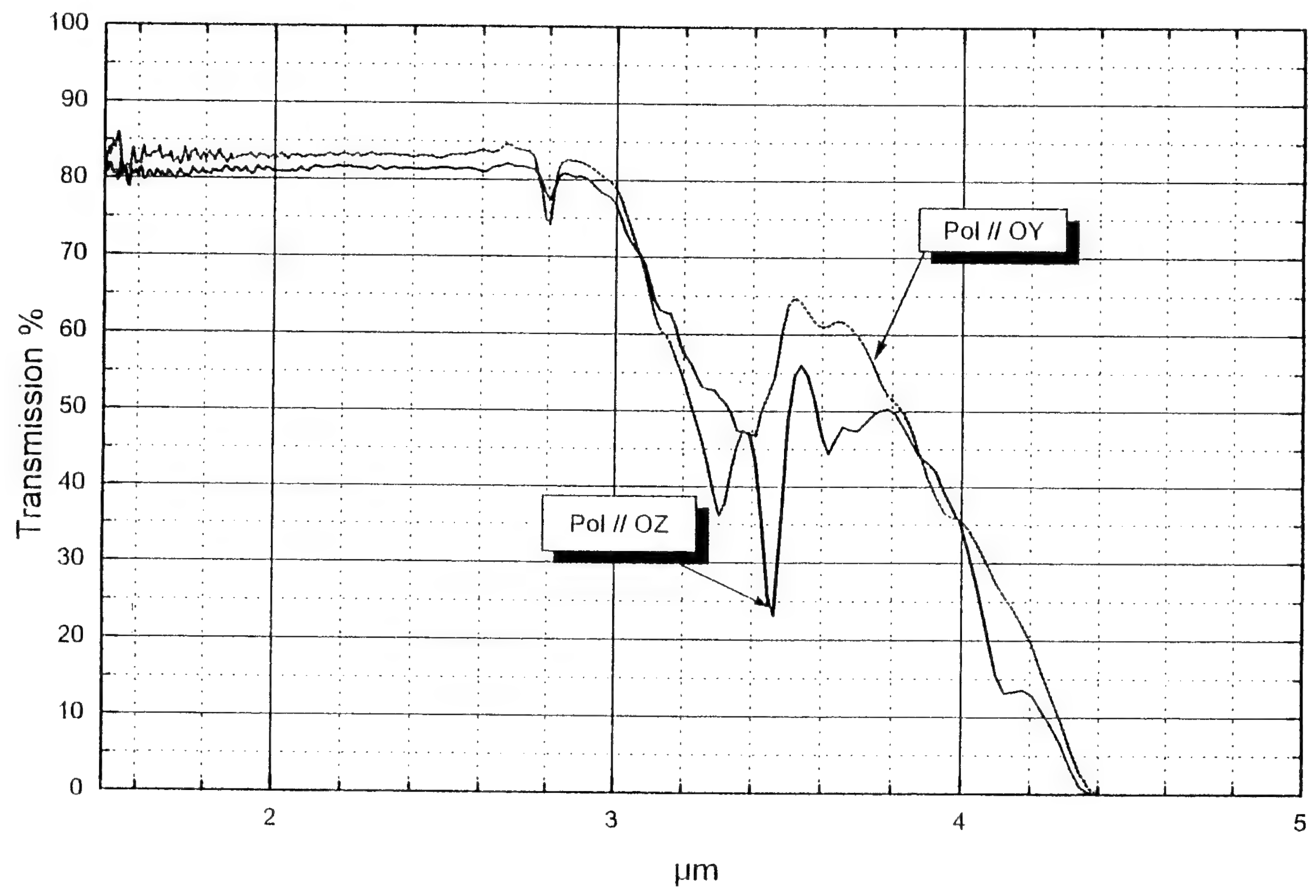
KTP N°4040109

ORIENTATION SUIVANT X



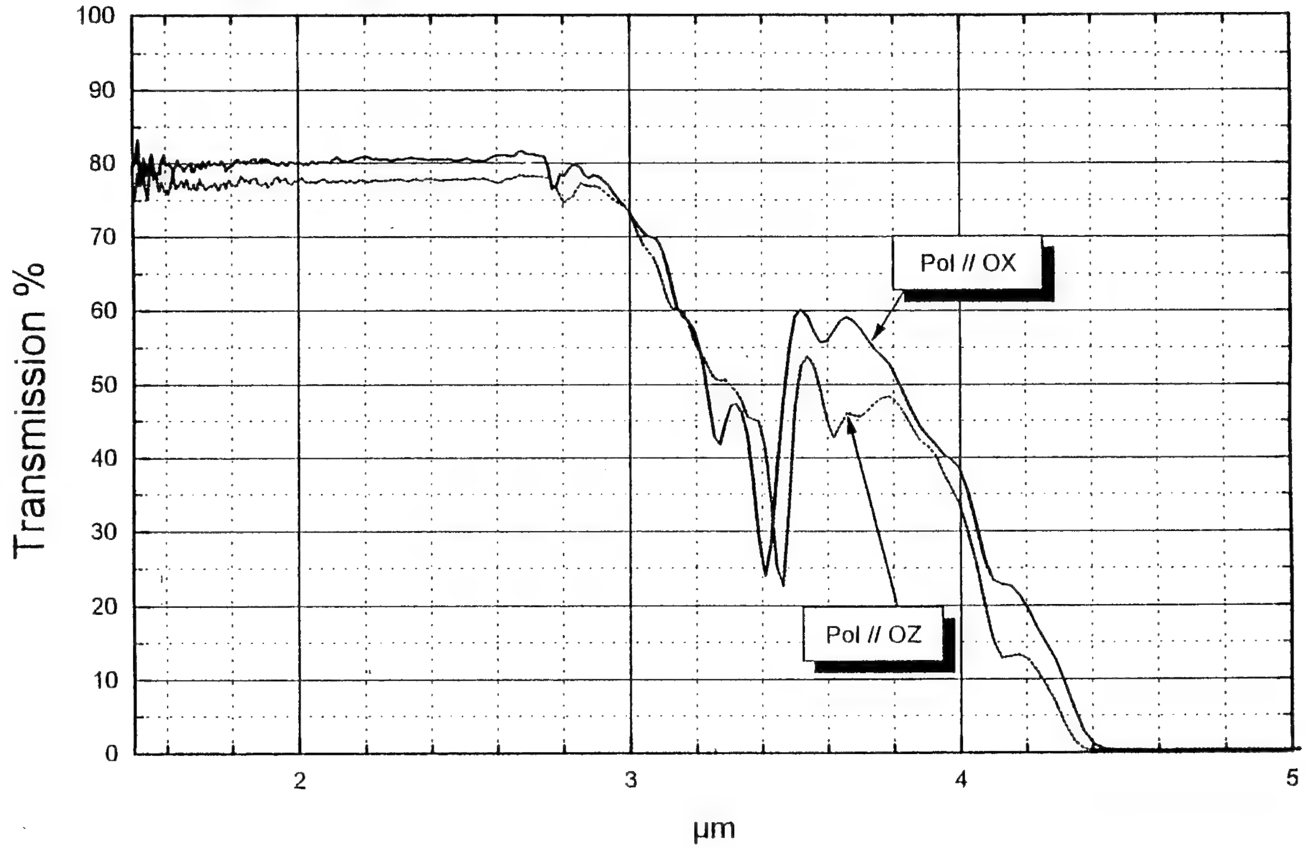
KTP N°4030720

Orientation suivant X



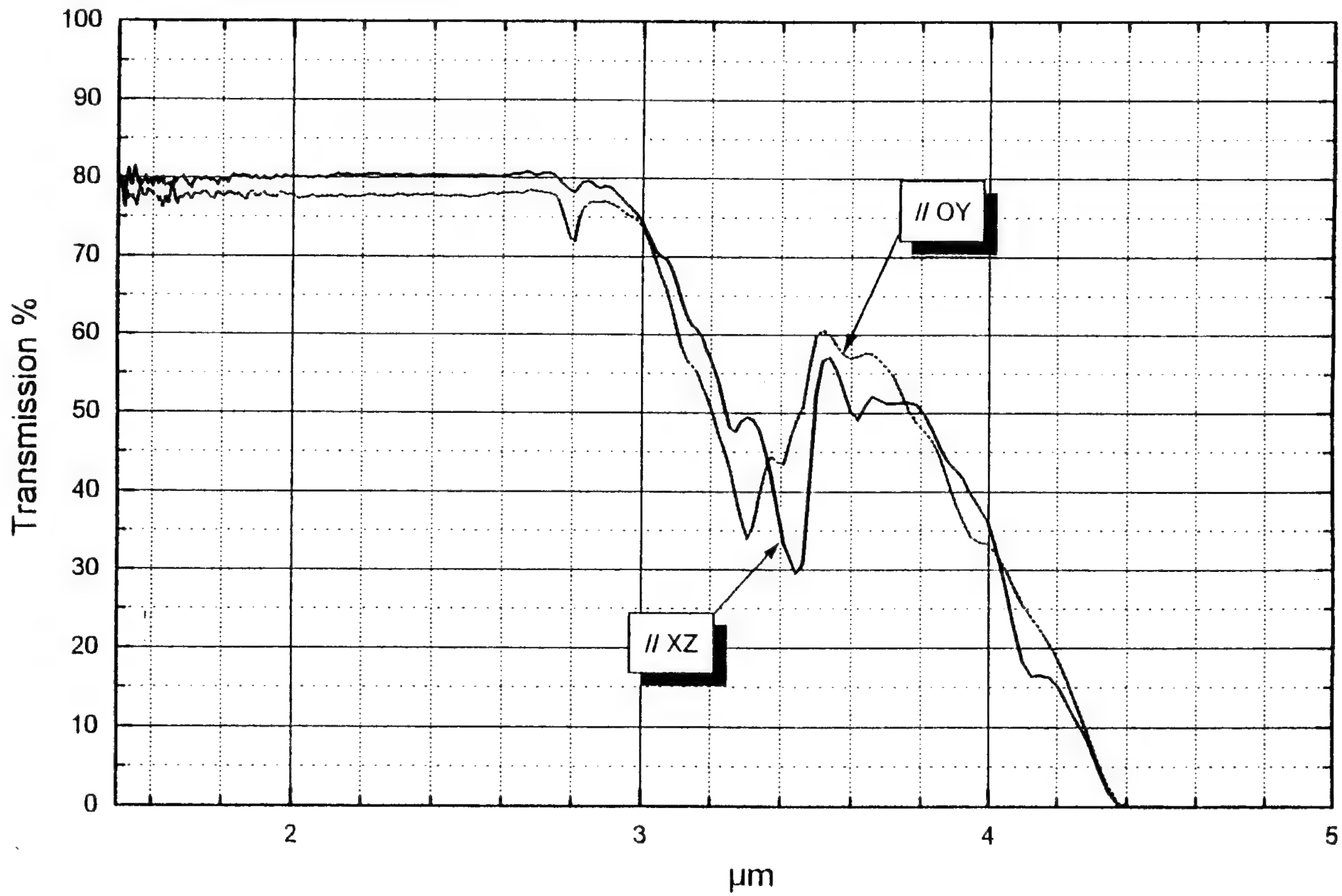
KTP N°4040222

Orientation suivant Y



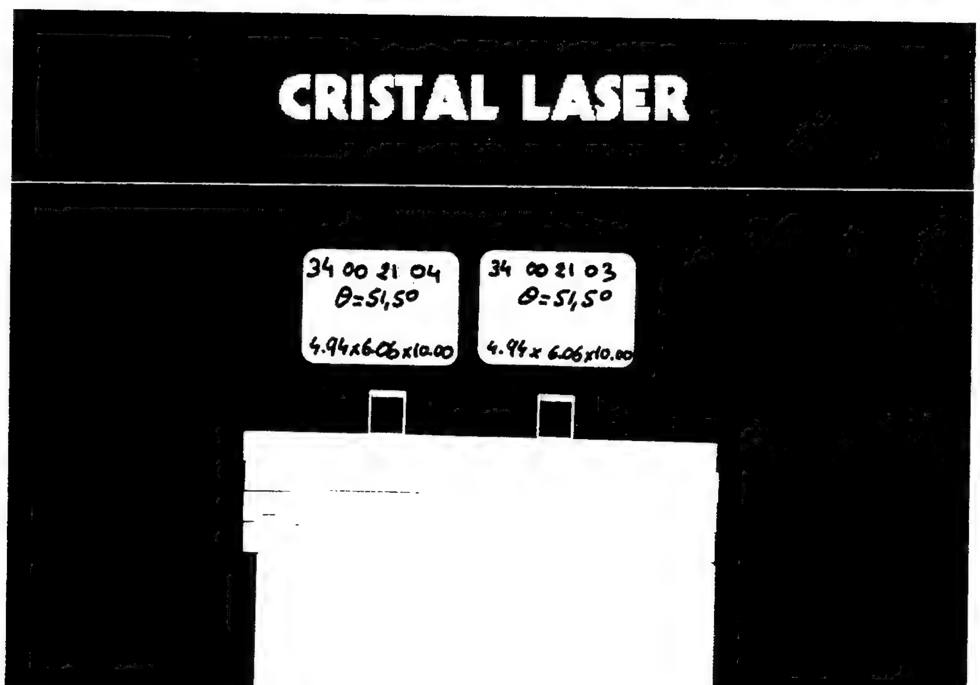
KTP N°3 40 02 04

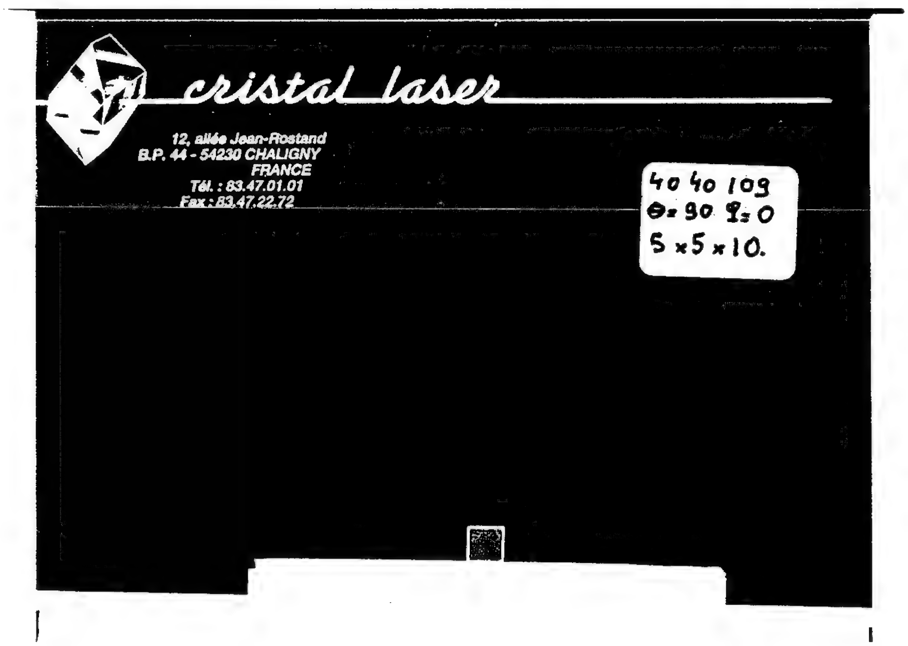
Orientation suivant XZ

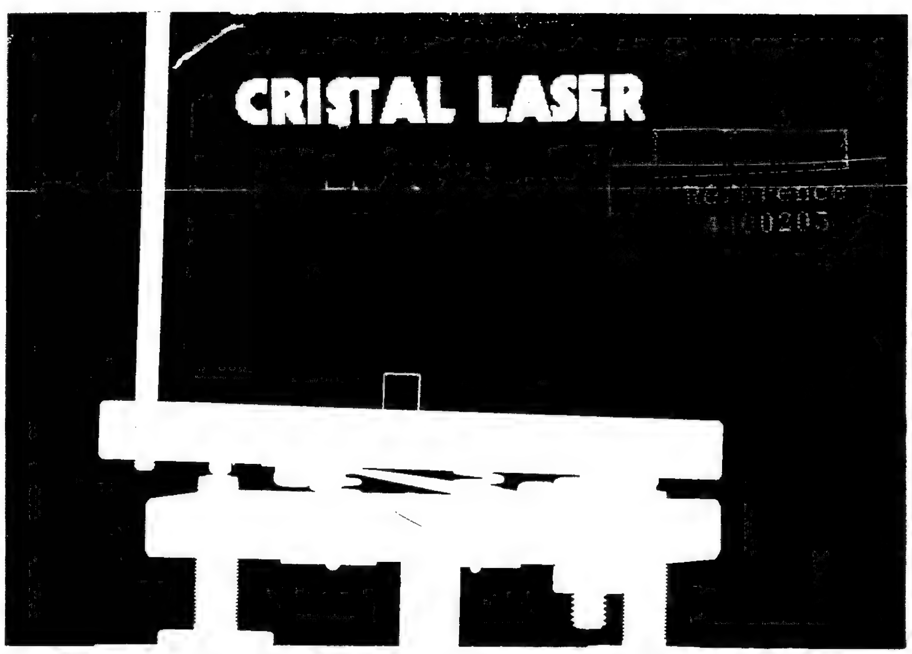


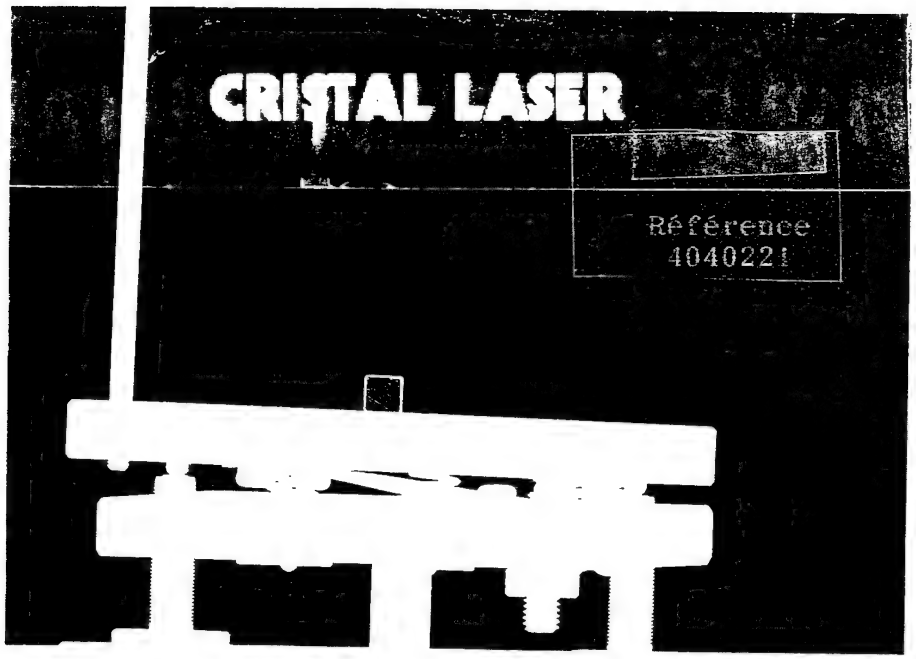
APPENDIX # 4

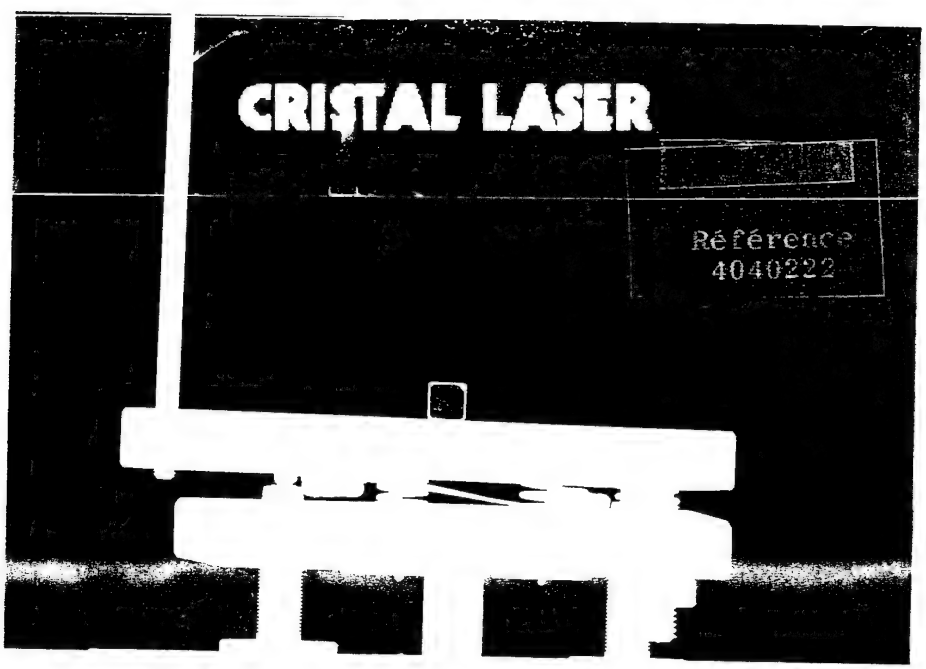
Homogeneity data


CRISTAL LASER
34 00 21 04
θ=51,5°
4.94x6.06x10.00
34 00 21 03
θ=51,5°
4.94x6.06x10.00


cristal laser
12, allée Jean-Rostand
B.P. 44 - 54230 CHALIGNY
FRANCE
Tél. : 83.47.01.01
Fax : 83.47.22.72
40 40 109
θ= 90 φ= 0
5 × 5 × 10.

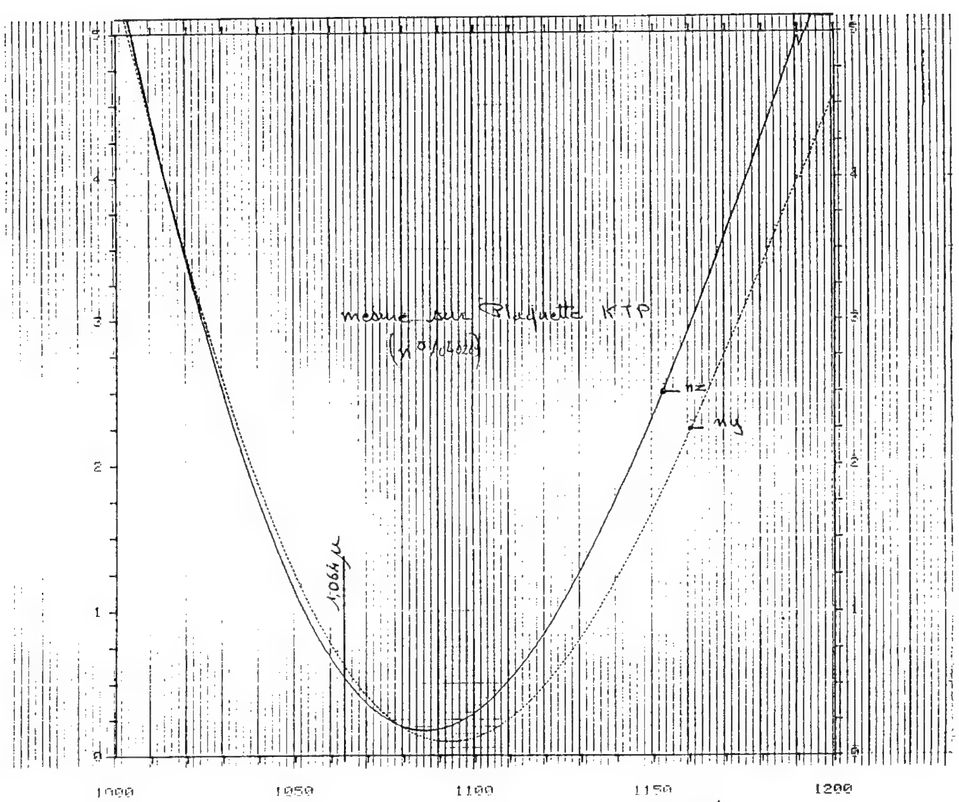

CRISTAL LASER


CRISTAL LASER
Référence
4040221

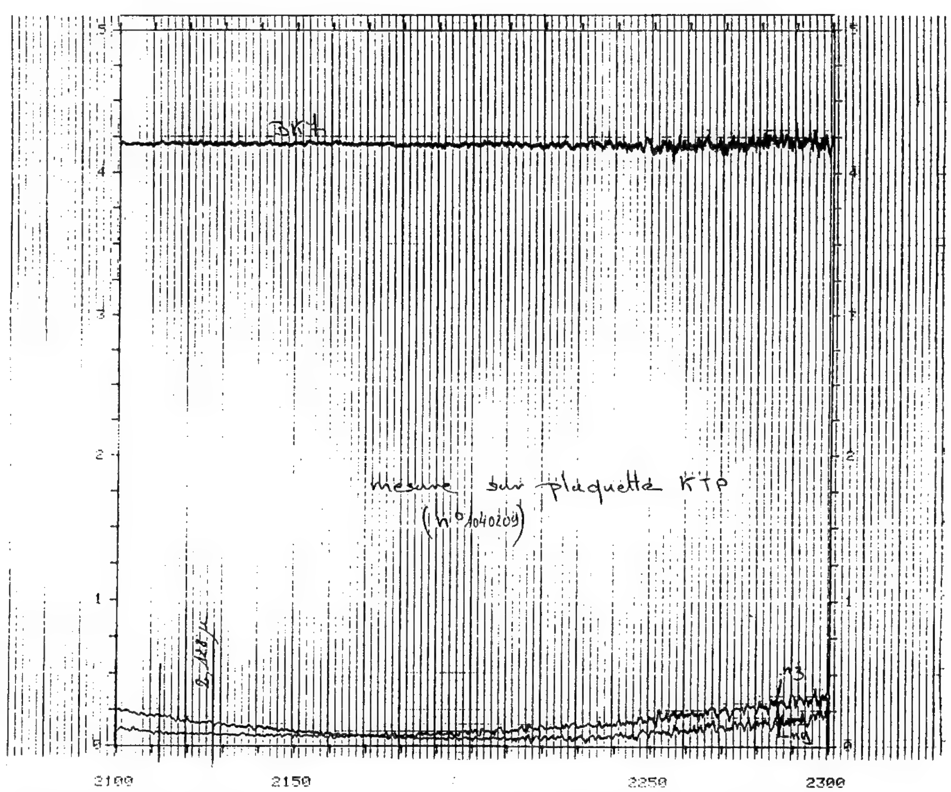

CRISTAL LASER
Référence
4040222

APPENDIX # 5

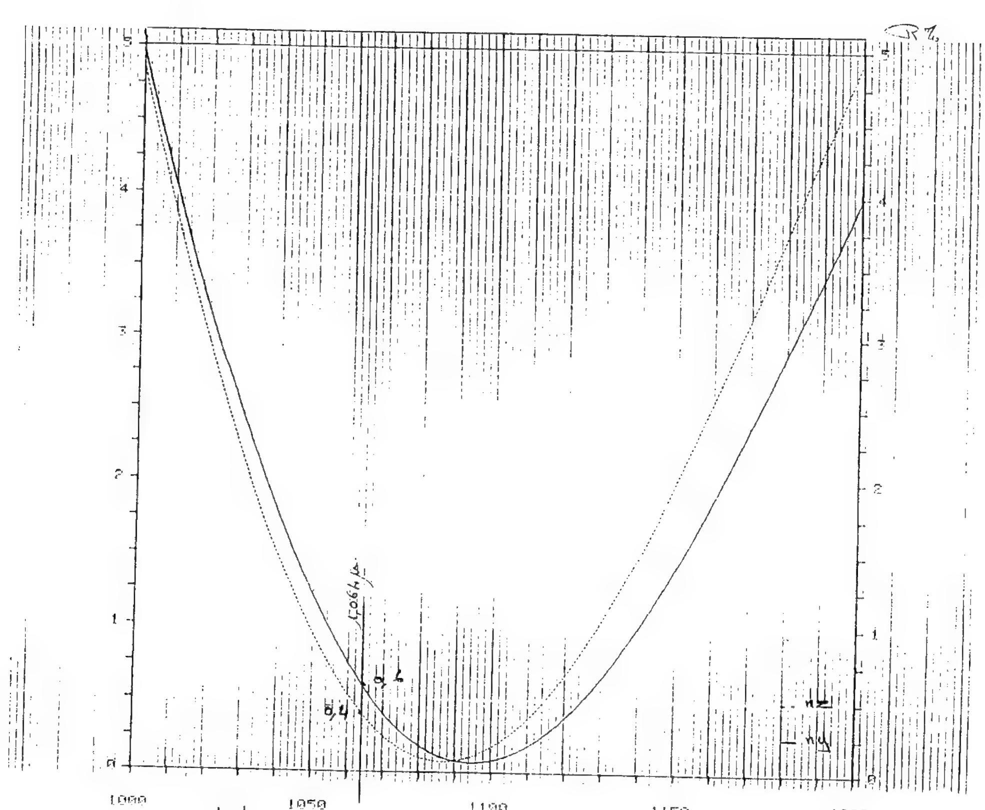
Reflection graphes of AR coatings
1.064+2.128 μm



(Calc no 1) 173/94/6 - R/60 - CRISTAL LASER - 24/1/95



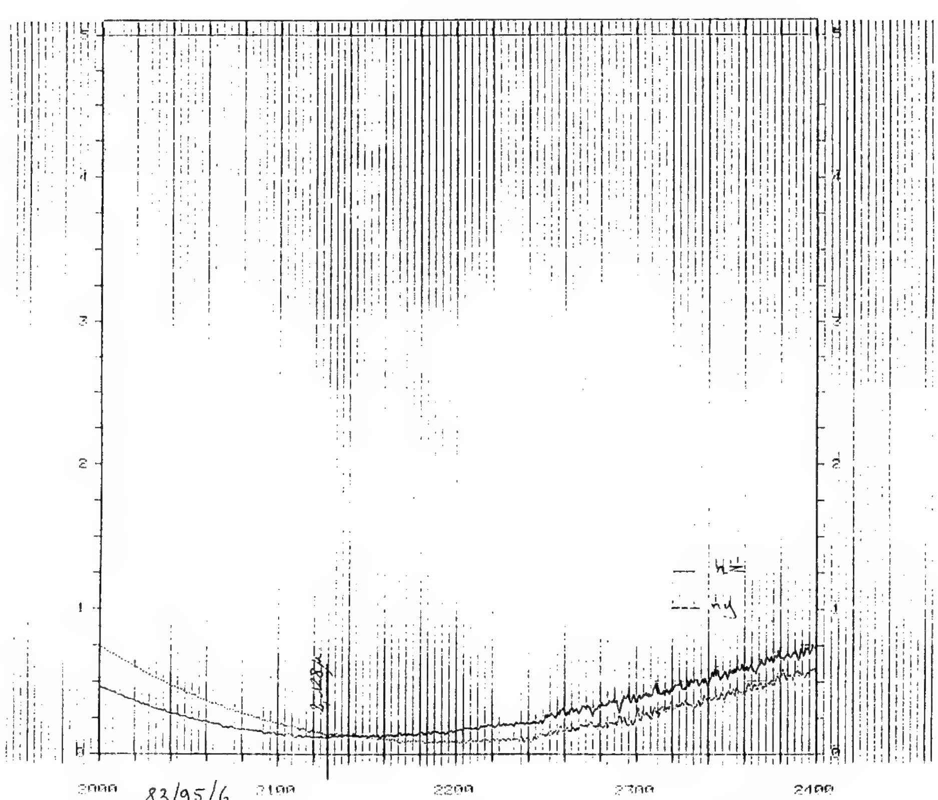
(cale n° 1) 1f3/94/6 - R/6° - CRISTAL LASER - 24/1/95



83/95/6

(Plaquette réf 1040209) R/5°

19/5/95



83/95/6

(Plaquette KTP n° 040209)

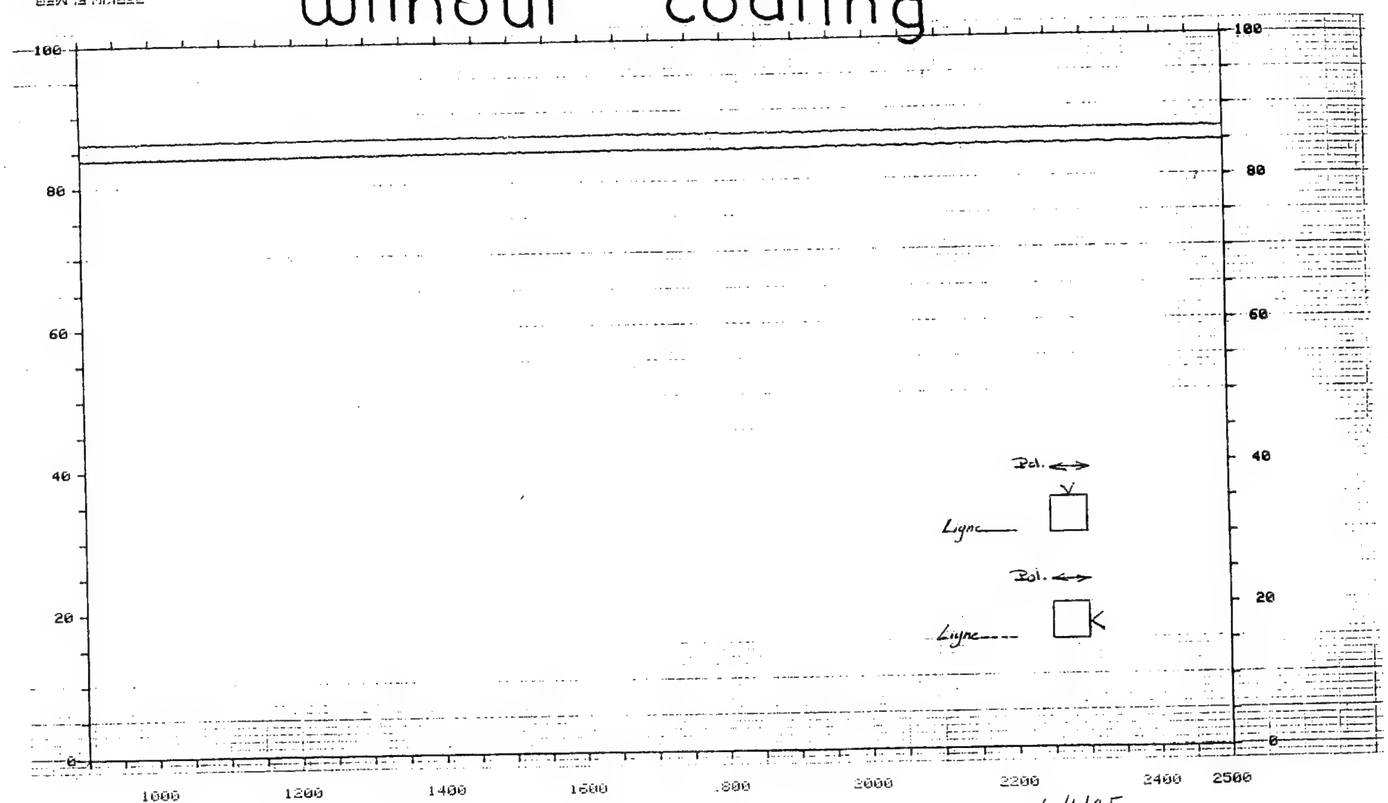
R/5°

19/5/95

APPENDIX # 6

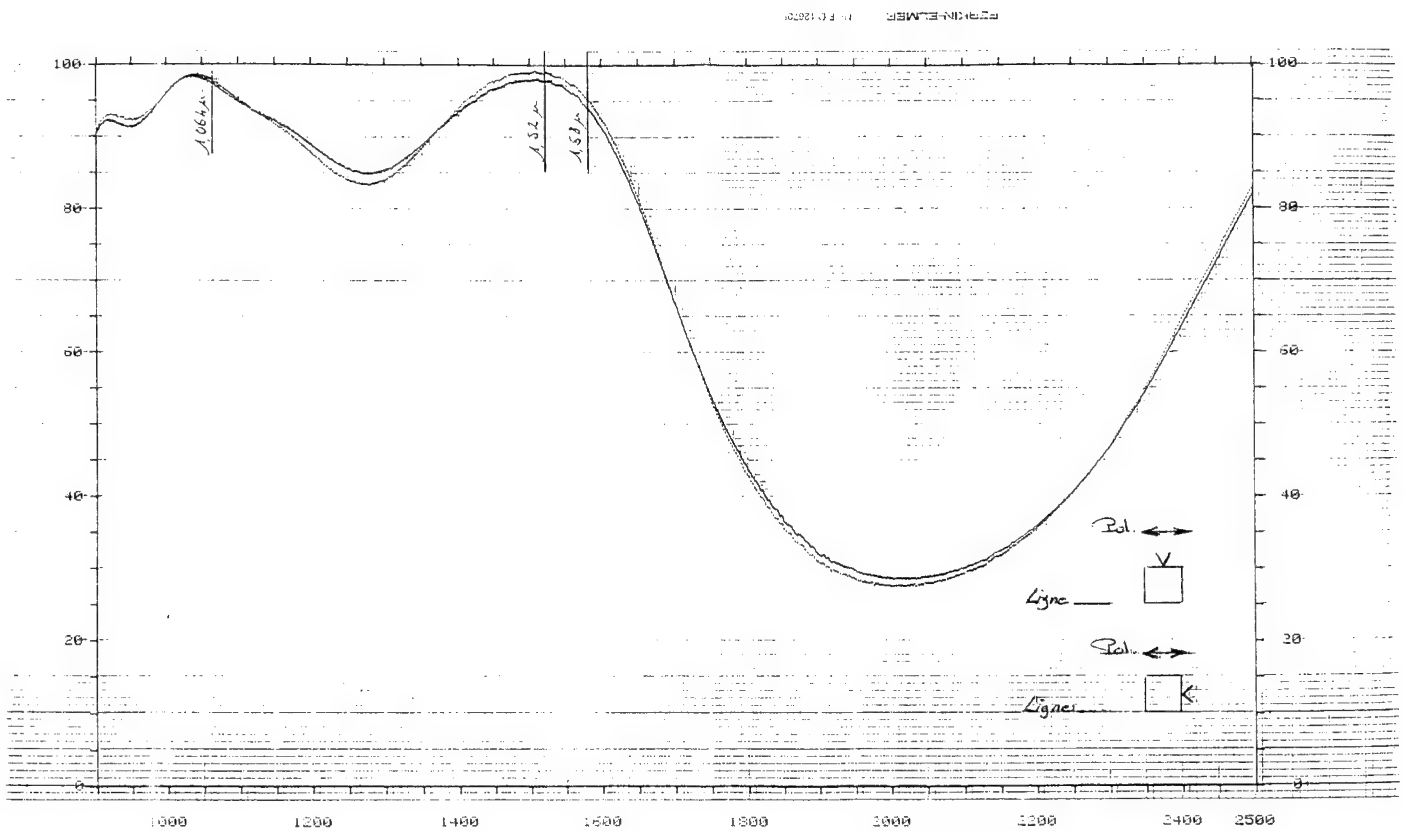
Reflection graphes of AR coatings
1.064 + 1.52-1.58 μm

without coating



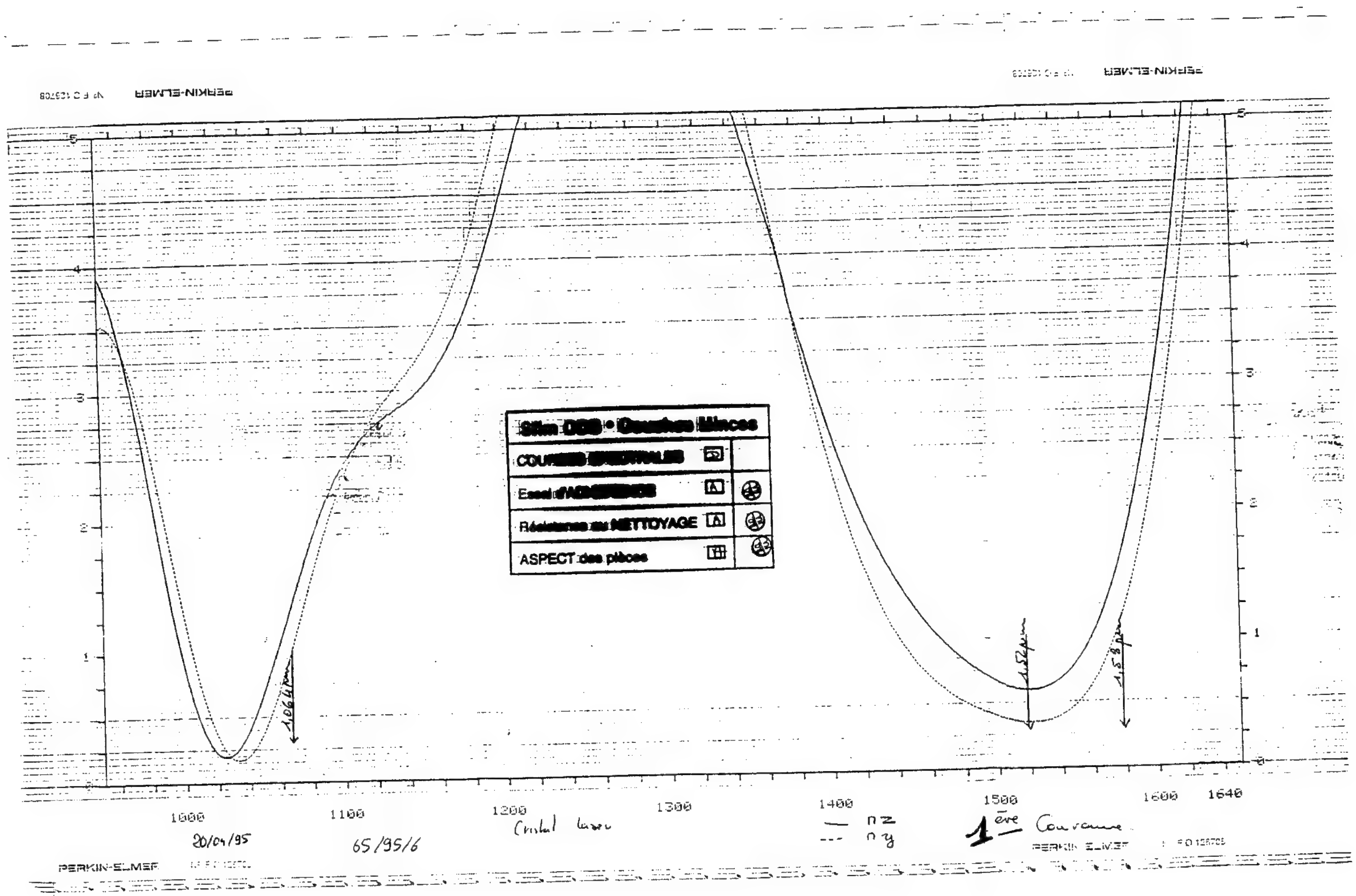
10/4/95

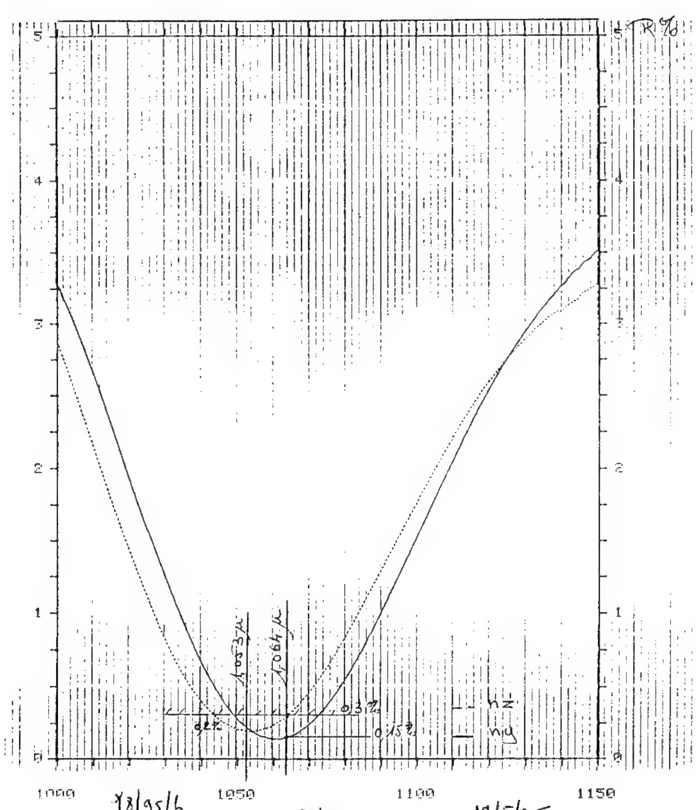
- KTP n° 4030724 - (avant trait.) T/0° en Polar.



KTP n° 4030724 (Après TAR 2 faces) T/0° en Polar [65-79/95/6] 19/5/95

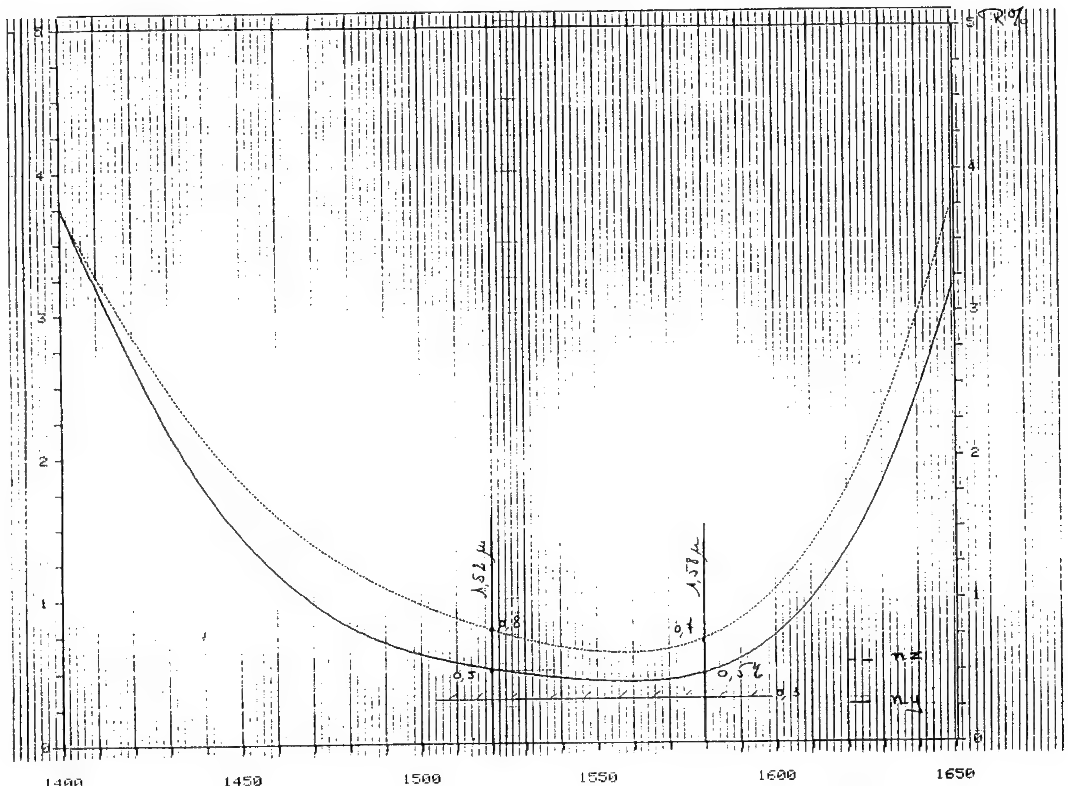
PERKIN-ELMER


PERKIN-ELMER
1000
1100
1200
1300
1400
1500
1600
1640
1,064 µm
1,52 µm
1,58 µm
ASPECT des pièces
20/04/95
65/95/6
Cristal laser
— nz
--- ny
1ère Couronne



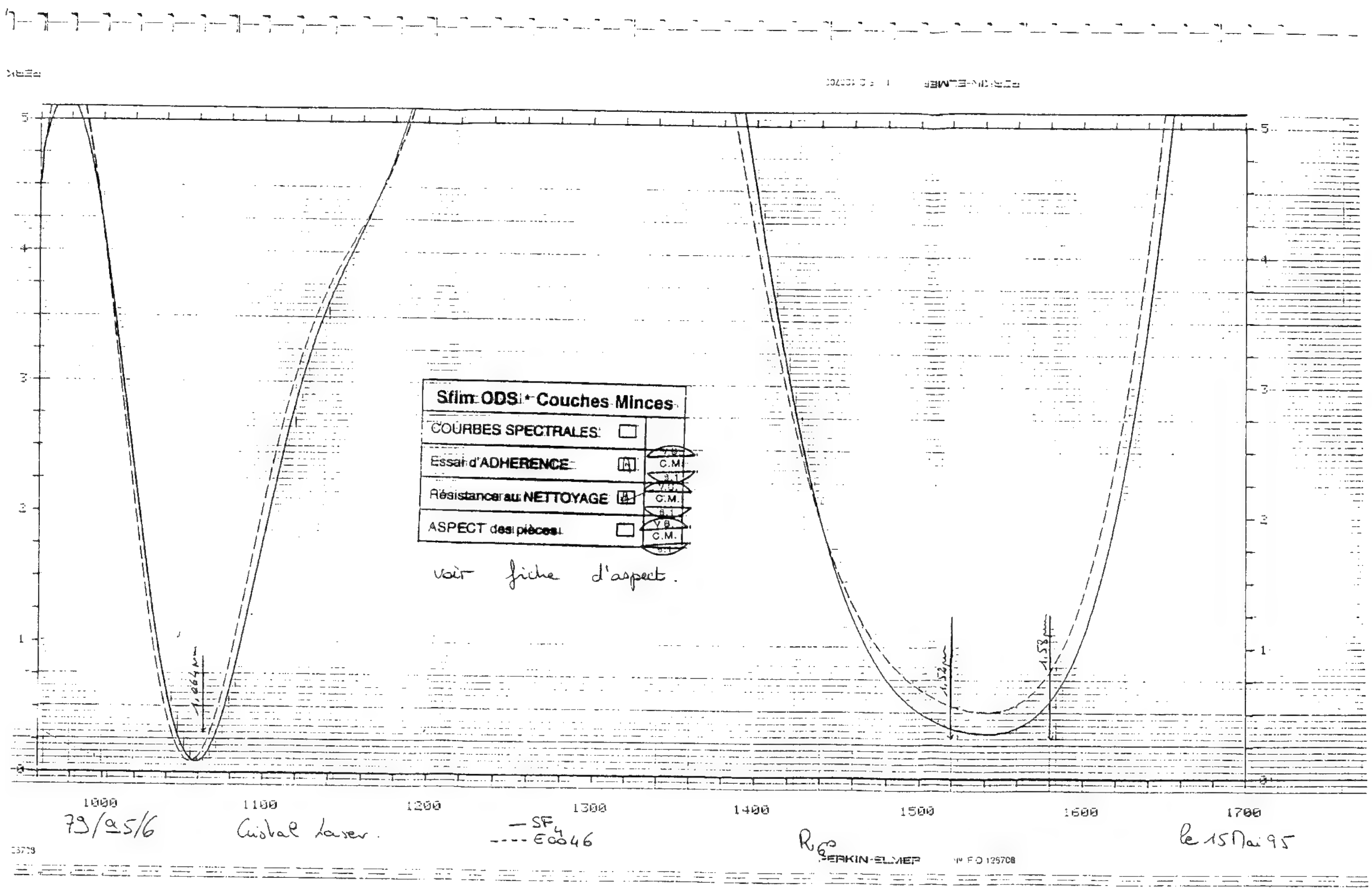
18/95/6 R/5° 19/5/95

(Plaquette réf. 4040208)



78/95/6 R/5° 19/5/95

(Plaquette ref 40402 08


Sfim ODS - Couches Minces
COURBES SPECTRALES
Essai d'ADHERENCE
Résistance au NETTOYAGE
ASPECT des pièces
C.M.
C.M.
C.M.
voir fiche d'aspect.
1,064μ
1,32μ
1,58μ
0
1
2
3
4
5
1000
1100
1200
1300
1400
1500
1600
1700
79/a5/6
Cristal laser.
— SF4
---- E0046
PERKIN-ELMER
le 15 Mai 95

TM 270795

ROPHOTOMETER
IR-Laser

SCAN/MANUAL Kristalle

%T/WL 11 pcs

2.00 NM/ /2

240 NM/MIN

0.5 S

DIS / 360.0 NM Coating

1 / 0.05 MIN

2 %T 2x HLC/642

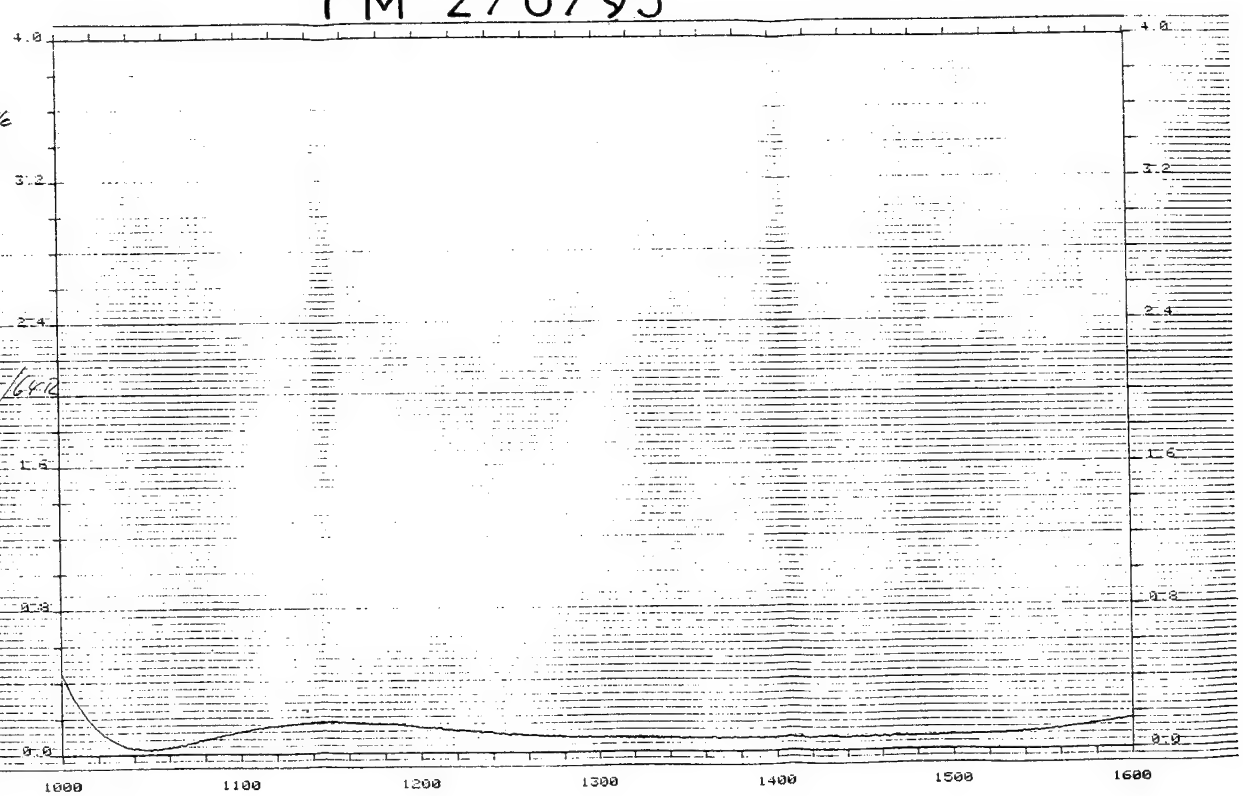
SERIAL / DASH 1

0.000 / 4.000

1000.0/1600.0

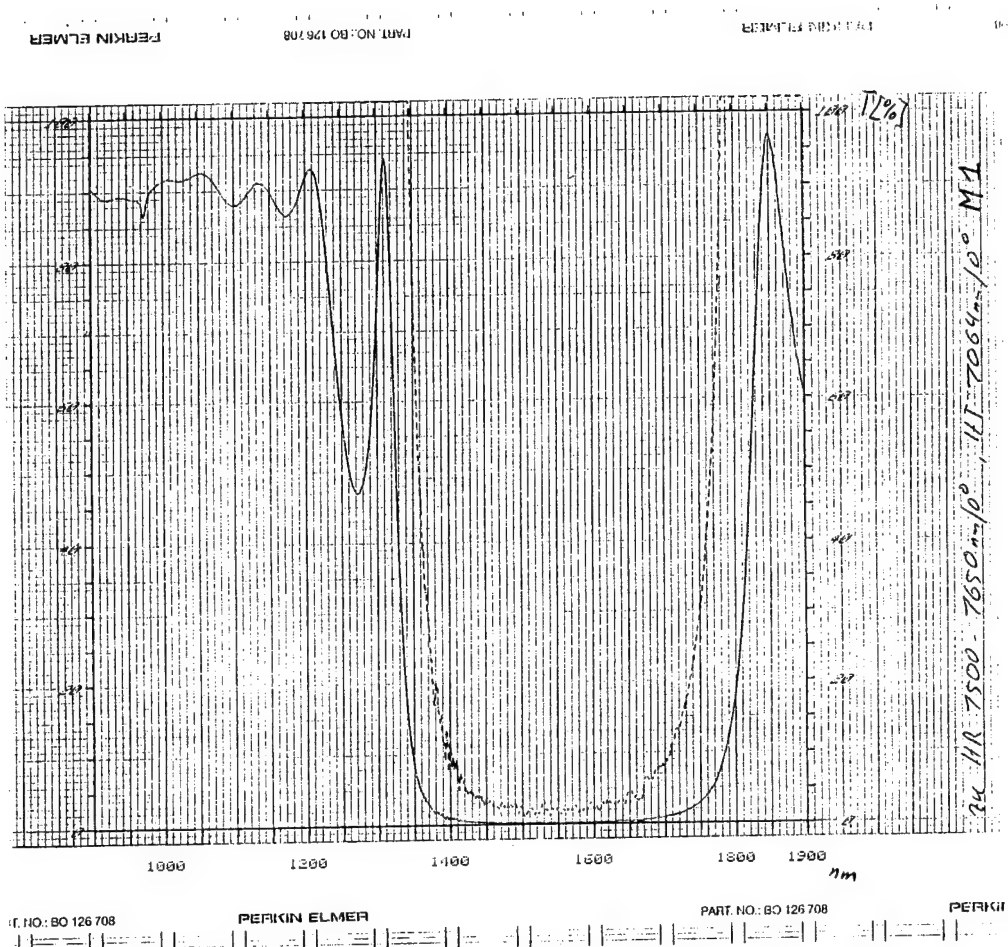
20 NM/CM

/METHOD



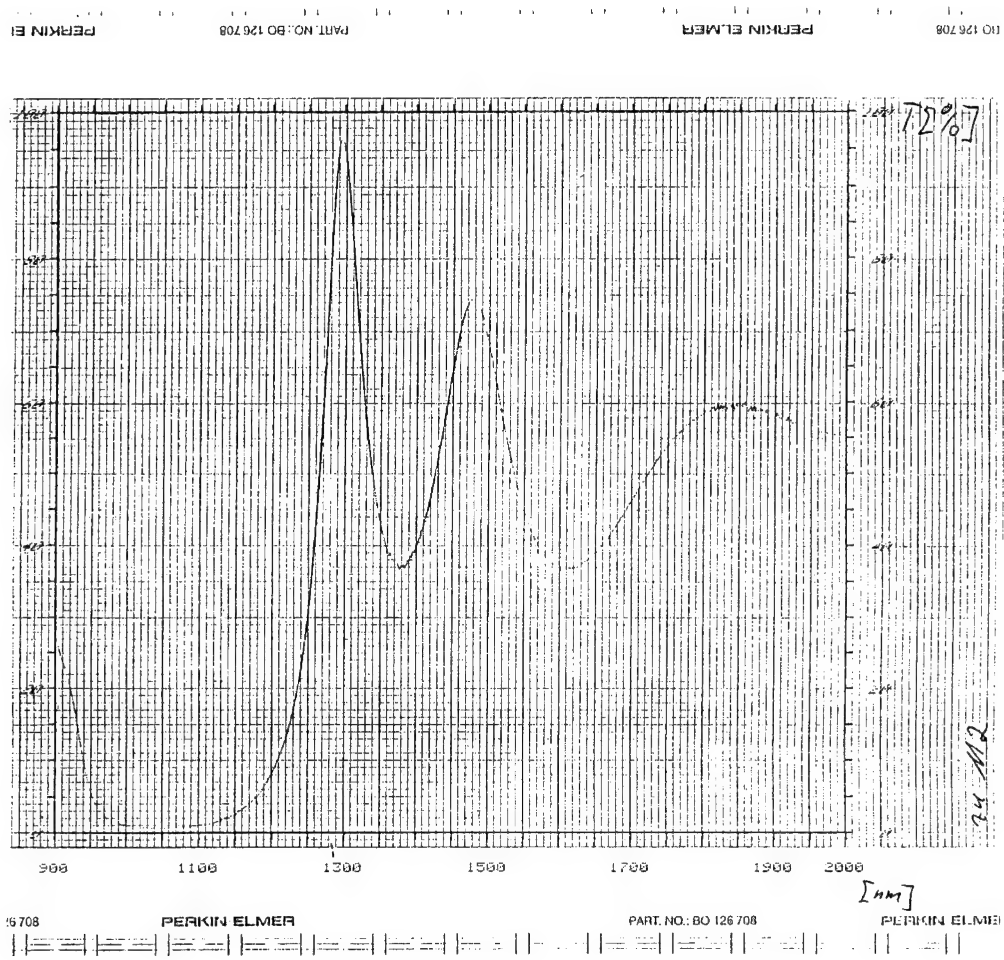
APPENDIX # 7

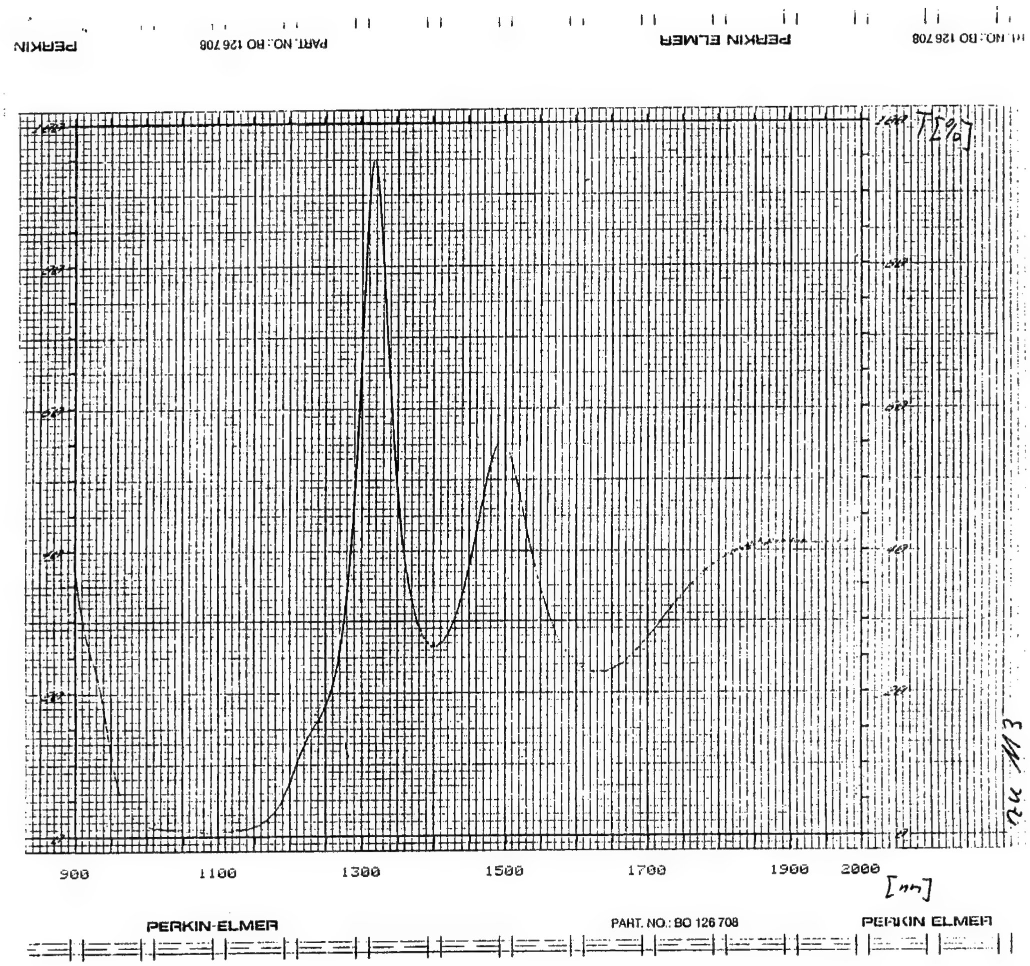
Transmission graphes of OPO mirrors

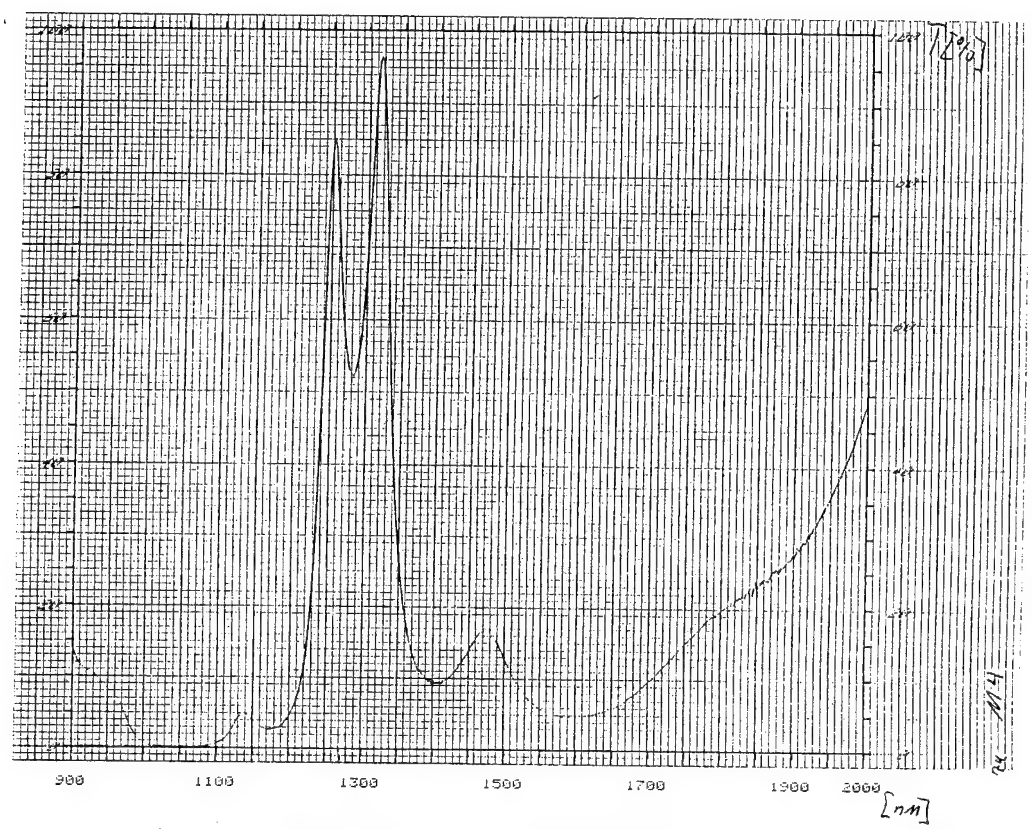


PERKIN ELMER
PART. NO.: BO 126 708
T[%]
2u HR 7500 - 7650nm/0°, HT 7064nm/0° M1
1000
1200
1400
1600
1800
1900
nm
PART. NO.: BO 126 708
PERKIN ELMER
PART. NO.: BO 126 708

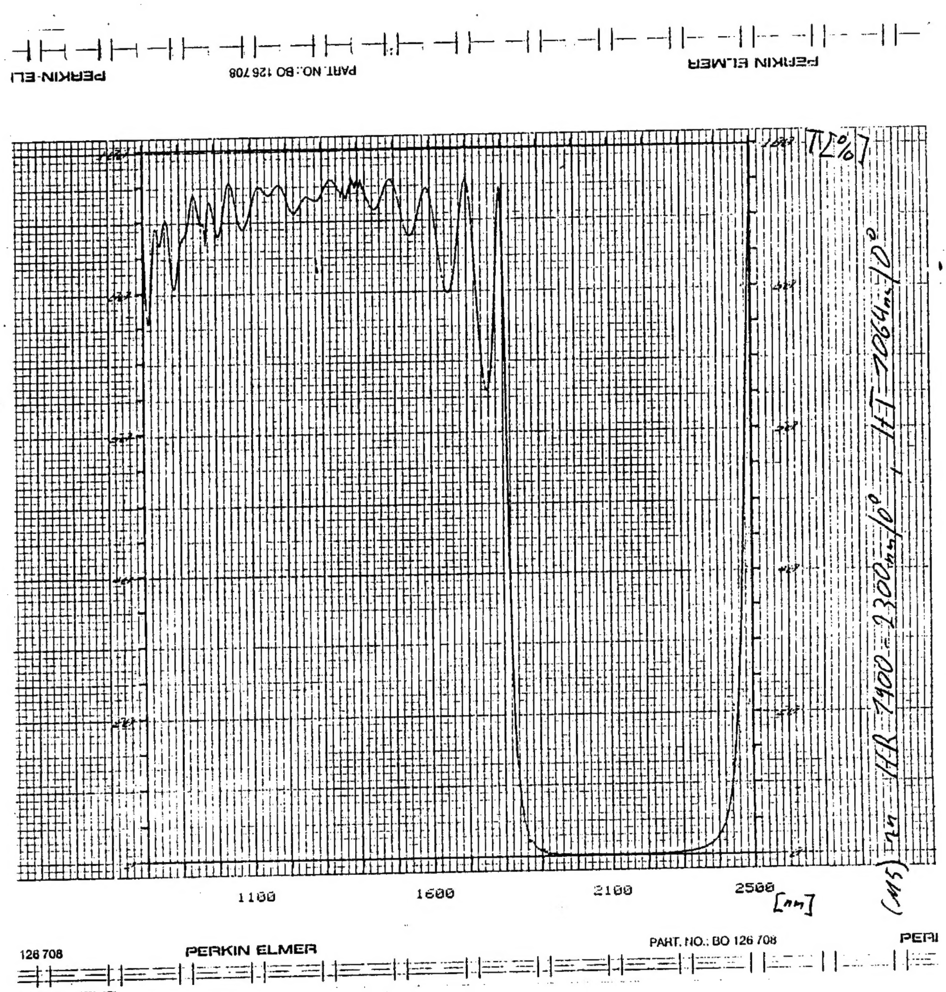
M 1


T [%]
900
1100
1300
1500
1700
1900
2000
[nm]
PERKIN ELMER
PART. NO.: BO 126 708


T [%]
900
1100
1300
1500
1700
1900
2000
[nm]
zu M3
PERKIN-ELMER
PART. NO.: BO 126 708
PERKIN ELMER

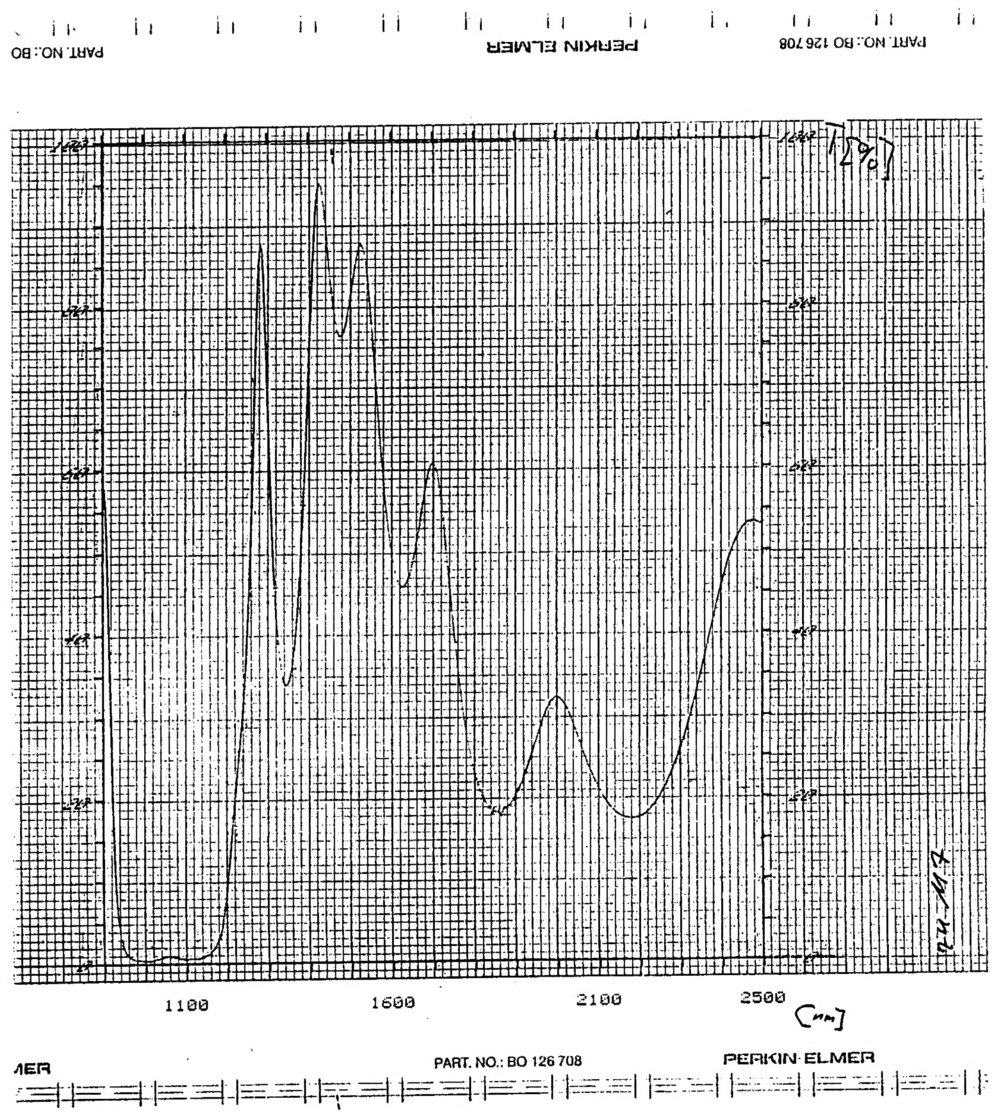


PART. NO.: BO 126 708 PERKIN ELMER

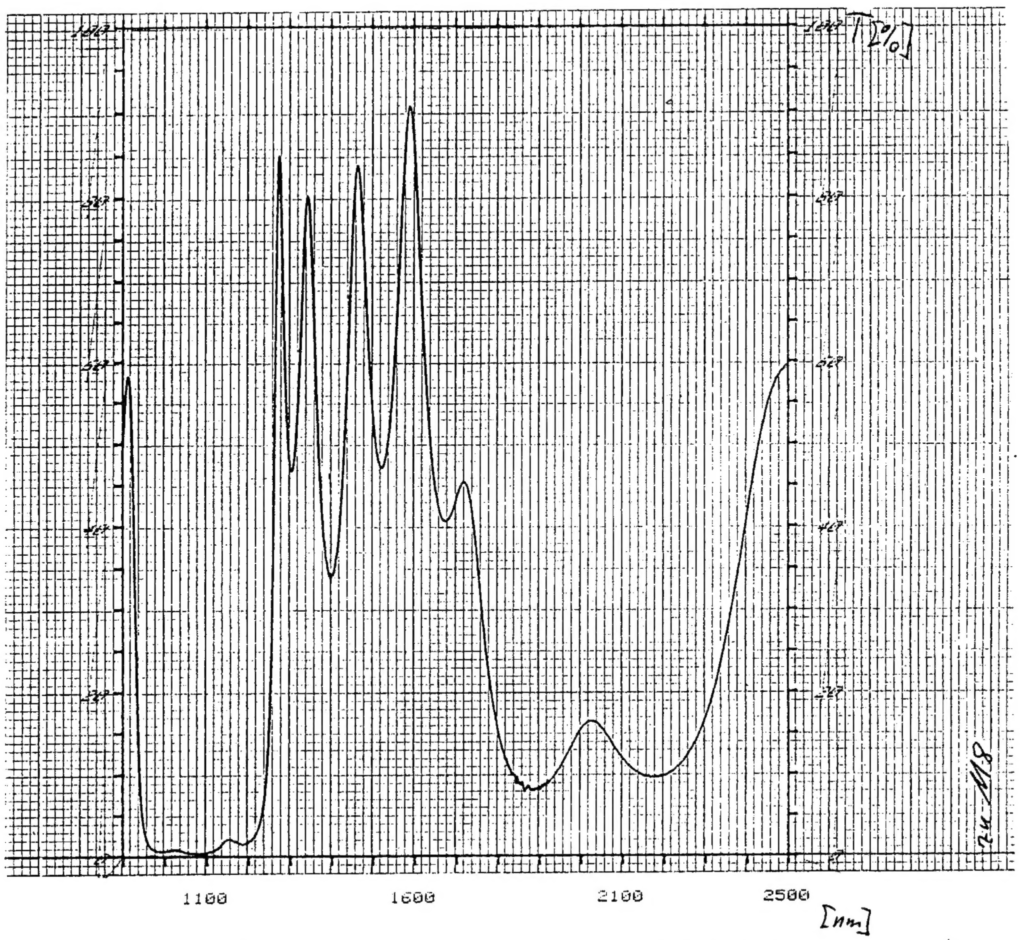


PERKIN-ELMER
PART. NO.: BO 126 708
PERKIN ELMER
T[%]
HR 1900-2300nm/0° + HT 1064nm/0°
(MS) 24
1100
1600
2100
2500
[nm]
126 708
PERKIN ELMER
PART. NO.: BO 126 708
PERI

[%]
1100
1600
2100
2500
[nm]
PERKIN-ELMER
PART. NO.: BO 126 708



[%]
1100
1600
2100
2500
[nm]
PART. NO.: BO 126 708
PERKIN·ELMER



100
80
60
40
20
0
[%]
1100
1600
2100
2500
[nm]